INTEROFFICE CORRESPONDENCE

139919

Piney River - August 20, 1968
OFFICE BLDG/TUBE RM# DATE

TO: Piney River Office

ATT'N. OF: Mr. J. F. Hopkins COPY TO: Mr. J. J. Fitzgerald

SUBJECT: Waste Acid Recovery

REFERENCE:

Our experience in waste acid recovery dates back to 1943 when we started work on a laboratory and semi-pilot plant scale to produce 70-72% H_2SO_4 from our waste end liquor. The recovered acid was used in laboratory equipment to react with our by-product apatite to produce superphosphate. Work was continued intermittently until early 1946 when Chemical Construction suggested the installation of a submerged combustion waste acid concentration pilot plant at Piney River.

This pilot plant was constructed and operated during 1947 and 1948. A complete report covering this entire operation has been issued. The process concentrated end liquor containing 22% H_2SO_4 and 11% $FeSO_4$ to a 50% acid solution in the primary stage. The production from this stage was filtered on a rotary filter to remove the precipitated $FeSO_4.H_2O$. The filtrate was then sent to a second stage where it was concentrated to 93% H_2SO_4. The ferrous sulfate monohydrate was processed through a sintering plant to produce iron oxide sinter and sulfur dioxide. Sulfur dioxide was used in an existing contact acid plant. The major difficulty encountered with this operation was in the second stage due to the corrosive effect of the hot 93% acid. The acid mist carried over with the water vapor and air also caused difficulty. It is our opinion that if this plant had been operated to produce a 72% acid less difficulty would have been encountered.

In 1952 we received information covering an acid recovery plant being operated by British Titan. They were using a newly developed high temperature karbate cubic heat exchanger for heating their acid using steam as the heat source. Using this information plus the information that we had obtained during the Chemical Construction program plus information on materials of construction which we obtained from suppliers of pumps, pipes, and valves, we designed a 10 ton per day pilot plant. This pilot plant was successfully operated and allowed us to produce 50% acid to prove by actual operation that the use of this acid would have no detrimental effect on the quality of our pigment. The success of this pilot plant led us to the decision that a 50 ton/day (100% H_2SO_4) plant should be installed producing 72% H_2SO_4. In 1958 working with Mr. W. B. Lambe of our Engineering and Construction Div., a plant estimate was prepared for the 50 TPD unit. This estimate showed that a maximum expenditure of $660,000 would be required. A report (PEO 147) covering this project was issued. The following paragraph was taken from this report referring to the data obtained in the 10 TPD pilot plant. "The continuous experience with this evaporating unit over a period of more than two years has provided considerable information on materials of construction, maintenance features, design factors relating to capacity, yields, steam usage, and other data pertaining to operating techniques. It is considered that the overall accumulation of technology which is reflected in the flowsheet of the proposed 50 ton plant is adequate for scale-up and engineering of the proposed unit."

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After review of the cost involved for the 50 ton plant, it was decided that we would install part of the proposed installation. In 1959 the present waste acid plant was installed and operation started. The plant order covering this installation was for $115,000. This plant has been successfully operated on a continuous basis. In July 1968 the plant produced 578 tons of 100% acid (as 55-57% H_2SO_4). This is an average production of 18.645 tons/day. Bound Brook uses a "Chemico" drum concentrator to concentrate their waste 70% H_2SO_4 to 93% H_2SO_4. We have reviewed this operation on two different occassions and have taken advantage of the "know how" they have developed. The proposed 70-72% acid plant is based on data which have been collected on a continuous basis since work was started on waste acid recovery. The present plant has been utilized to test various materials of construction for their resistance to hot sulfuric acid. We have also endeavored to keep up to date with all equipment manufacturer's experience on all concentration of sulfuric acid. We feel that the plant which is shown on the attached flowsheet will represent the best know how available in the industry today. I am also attaching a flowsheet which was submitted to our Engineering and Construction Division for their 1957 estimate.

We have prepared several gallons of 72% acid in the pilot plant. This acid has been used in the development laboratory to prepare pigment from Piney River ilmenite. Pigment quality has been equal to the control where new acid was used. The pilot plant runs were also used to check chemical analysis of the various acids and to prepare a material balance to show where the various impurities left the system. Reports showing the chemical analysis and the material balance have been issued to development and other departments concerned with waste disposal at Piney River. We have five (5) gallons of 72% H_2SO_4 at Piney River if any additional work is desired.

The proposed 72% H_2SO_4 plant has been designed with enough flexability to allow experimental runs which will furnish data for additional expansion.

Stephen A. Lamanna

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SAL/kcw

ATTACHMENT:

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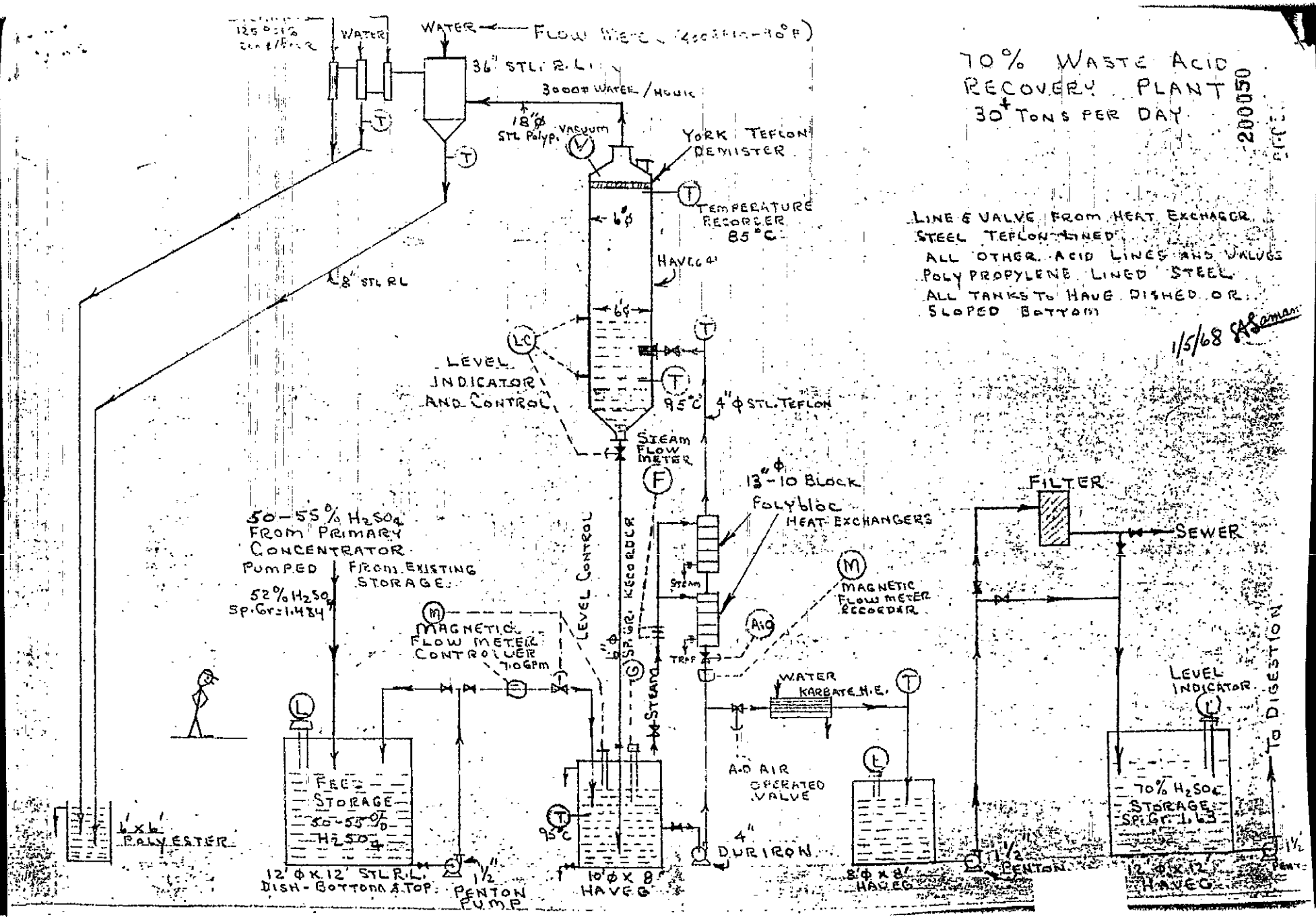
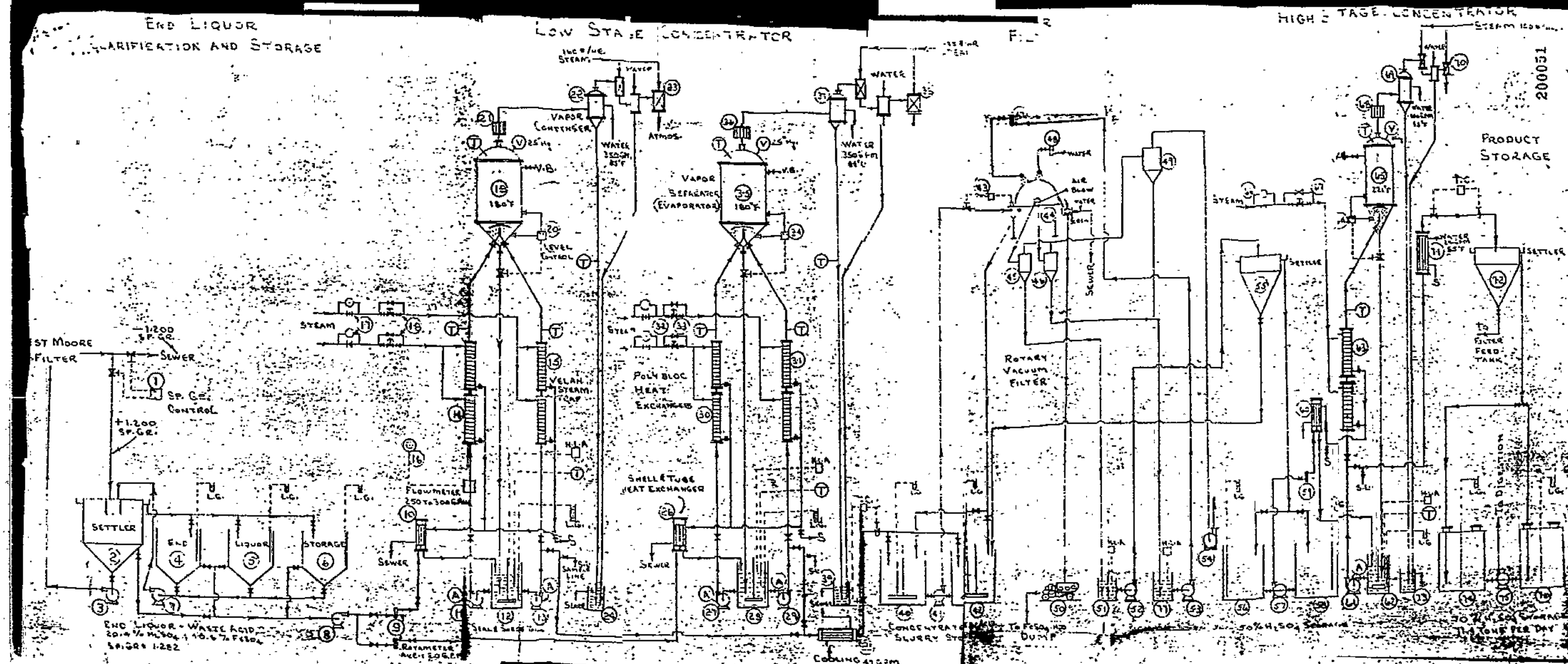

70% WASTE ACID RECOVERY PLANT
30+ TONS PER DAY
200050
LINE & VALVE FROM HEAT EXCHANGER STEEL TEFLON LINED
ALL OTHER ACID LINES AND VALVES POLY PROPYLENE LINED STEEL
ALL TANKS TO HAVE DISHED OR SLOPED BOTTOM
1/5/68
WATER
WATER ← FLOW METER (ROOM TEMP 70°F)
36" STL. R.L.
3000# WATER / HOUR
18"ø STL. Polyp.
VACUUM
YORK TEFLON DEMISTER
TEMPERATURE RECORDER 85°C
6"ø
HAVEG
8" STL. R.L.
6"ø
LEVEL INDICATOR AND CONTROL
95°C
4"ø STL. TEFLON
STEAM FLOW METER
13"ø - 10 BLOCK
POLYBLOC HEAT EXCHANGERS
50-55% H2SO4 FROM PRIMARY CONCENTRATOR PUMPED FROM EXISTING STORAGE
52% H2SO4 Sp. Gr. 1.484
MAGNETIC FLOW METER CONTROLLER 7.0 GPM
LEVEL CONTROL
6"ø
Sp. GR. RECORDER
STEAM
MAGNETIC FLOW METER RECORDER
TRAP
WATER
KARBATE H.E.
A-O AIR OPERATED VALVE
4" DURIRON
FREE STORAGE 50-55% H2SO4
95°C
6'x6' POLYESTER
12'ø x 12' STL. R.L. DISH-BOTTOM & TOP
1½" PENTON PUMP
10'ø x 8' HAVEG
8'ø x 8' HAVEG
FILTER
SEWER
LEVEL INDICATOR
70% H2SO4 STORAGE Sp. Gr. 1.63
TO DIGESTION
1½" PENTON
12'ø x 12' HAVEG



H.L.A. – High Level Alarm. (T) Temperature – L.G. Level Gauge – (V) Vacuum – S. Sewer – V.B. Vacuum Breaker (A) Ammeter – T.C. Temperature Controller (1) Specific Gravity Controller to insure delivery of all 1.200 Sp.Gr. Waste Acid to Recovery Plant – (2) End Liquor Settler – 16'-0" Φ × 5'-0" Cyl. Section – Cone Bottom, 8'-0" High – Steel Rubber Lined (3)(7) Sludge Transfer Pumps – LaBour Pump – Lead Casing & Impeller – D.D.S. or Worthite Capacity 50 GPM. (4)(5)(6) End Liquor Storage Tanks – Steel Rubber Lined 12'-0" Φ – Dished Bottom and Top – Approx. Total Vol. 12,000 Gals. (8) End Liquor Feed Pump – LaBour Pump – R-55 Casing & Impeller or Worthite – Cap. 60 GPM. (9) Two (2) Rotameters – Lead Heads – Hastelloy B Floats. Range 3 to 30 G.P.M. (10)(27) Karbate Shell & Tube Heat Exchangers to preheat End Liquor using Condensate from Polybloc – 87 sq. ft. unit required. (11)(13)(27)(29)(61) Circulating Pumps – Durimet 20 – Cap. 300 G.P.M. – Five (5) Pumps Required (Durimet 20, 4" suction, 3" discharge) Other Pump Materials – Hastelloy D, Haveg 41, Acid Glass (12)(29) Seal & Surge Tank – 10' Φ × 10' Steel – 4½" Acid Brick with Lead, Rubber or Pyroflex – One Blade Haveg Agitator – 20 to 30 RPM (14)(15)(30)(31)(63) Polybloc Heat Exchangers (The Carbone Corp., Boonton, N.J.) Ten (10) 12 Block Units Required – Each Unit = 66 Sq. Ft. – All Units Equipped with Velan Steam Traps (16) Foxboro Magnetic Flowmeter (Liquor = 22.3% FeSO4·H2O, 36.6% H2SO4, [illegible]% H2O, Sp. Gr. 1.71, Temp. 180°) Range 100 to 400 G.P.M. (17)(32)(66) Recording Steam Flowmeter with Integrator. Range 0 to 6000 #/Hour. Five (5) Units Required. (18)(33)(67) Pressure Regulator & Controller. Range 0 to 120 PSIG. Five (5) Units Required (19)(35) Vapor Separators (Evaporators) Cyl. Section 9'-0" Φ × 10' H., Dished Head / 60° Slope Cone, Steel – Acid Brick with Lead or Rubber. Alt. Haveg or Steel Lead Clad (20)(34)(69) Level Controllers to Control Levels in Evaporators (21)(36)(68) Karbate Entrainment Separators (22)(37) No. 6 S&K (Schutte-Koerting) (14' Φ) Counter-Current Condensers – Steel Rubber Lined – Capacity 7000 #/Hour Water Vapor @ 5" Hg Abs. Using 350 G.P.M. 85° H2O – Tail Leg Steel Rubber Lined – Leg in Seal Tank – Lead (23)(38) 2" S2 – S&K Steam Jet Exhauster, Haveg Nozzle & Body with Bronze Steam Chest and Stainless Steel Integral Strainer – Steam 160 #/Hour @ 120 PSIG – Air Cap. 39 #/Hour (24)(39) Tail Leg Tank 10' Φ × 8'-0" H. – Steel Rubber Lined (25) Karbate Shell & Tube Heat Exchanger to Cool Concentrated Slurry – 87 Sq. Ft. Required (40)(42) Concentrated Acid Slurry Storage 12'-0" Φ × 12'-0" H. Steel Rubber Lined Tanks – One (1) Blade Steel Rubber Covered Agitator – 30 R.P.M. (41) Filter Feed Pump – Durimet 20, Lead Casing & Impeller or R-55. Cap. 30 GPM.

(43) Filter Bowl Level Control (44) 8'-0" Φ × 8'-0" Face Rubber Covered Rotary Vacuum Filter – PVC Filtrate Tubes – Open Trough for Inspection – Carpenter 20 Doctor Blade – Fog Wash Nozzles – Carpenter 20 – Cake Screw Conveyor (45) 24" × 48" Steel Rubber Lined Filtrate Receiver – Pick Up & Weak Acid Wash (46) 24" × 48" Steel Rubber Lined Filtrate Receiver – Water Wash & Dry (47) Rotameter for Weak Acid Wash – Lead Heads – Hastelloy B Float – Range (H2O) 1 to 10 G.P.M. (48) Std. Rotameter for Water – Range 1 to 10 G.P.M. (49) 24" × 72" Steel Rubber Lined Condenser (50) FeSO4·H2O Dump Truck with Acid Proof Body (51) Strong Liquor Filtrate – 3' Φ × 5' (265 Gals.) Steel R.L. (52) LaBour R-55 Pump Cap. 30 GPM (77) Weak Acid Filtrate 3' Φ × 5' Steel R.L. (53) LaBour R-55 Pump Cap. 20 G.P.M. (54) #7 Nash Vacuum Pump – 300 CFM – 20" Hg (55) Settler – Steel R.L. 12' Φ × 5' – 45° Cone Bottom [illegible] (56)(58) Filtered Acid St. 10'-0" Φ × 10' Steel R.L. Tank (57) Durimet 20 – H.S. Feed Pump – Cap. 20 G.P.M. (59) Rotameter – Lead Heads – Hastelloy B Float – Range 2 to 20 G.P.M. H2SO4 Sp.Gr. 1.421 (60) Shell & Tube Karbate Heat Exch. to Preheat Acid – 87 Sq. Ft. (62) Seal & Surge Tk. 8'-0" Φ × 10'-0" Ht. Steel – Asbestos – Lead – 4½" Acid Brick (65) Vapor Separator (Evaporator) 6'-0" I.D. × 8'-0" I.Ht. – 60° Slope Cone Bottom – Dished Head Steel Lead Clad or Steel – Lead – Acid Brick. (62) Equipped with 1 Blade Haveg Agitator – 20 RPM (69) No. 4 (14" Φ) S&K Barometric C.C. Condenser – Steel R.L. 2500 # H2O Vapor/Hr. – 25" Hg – 100 GPM H2O 85°F (70) 1½" S-2 S&K Steam Jet Exhauster – Construction as Items 23 & 38 – 100 # Steam/Hour – Cap. 21 # Air/Hour (71) Karbate Shell & Tube H.E. to Cool 70% H2SO4 – 87 Sq. Ft. Required (72) Strong Acid Settler – Steel Hypalon Rubber Lined – 10' Φ × 4' with 45° Slope Cone Bottom (73) Tail Leg – 4' Φ × [illegible] Polyester Tank (74)(76) 70% H2SO4 Stor. Tks. – Steel – 12'-0" Φ × 14'-0" (75) 70% H2SO4 Transfer Pump Cap. 80 G.P.M. LaBour R-55 or Durimet 20. Carbo-Korez Cement Used with All Acid Brick – All Acid Containing Tanks to be Covered – Cold Acid – Steel Rubber Lined – Hot Acid Haveg 41 or Lead Clad Steel. Piping – Valves: Cold End Liquor <60°C Saranlloy Lined Steel Pipe – [illegible] Valves – Saranlloy Lined and Diaphragms – Hot End Liquor 90°C Haveg 41, Lead Lined Steel, Heavy Wall Lead Pipe, Valves Haveg 41 – Lead or Glass – Low Stage Conc. – [illegible] (50–90°C) Haveg 41, Heavy Wall Lead or Epoxy Pipe – Valves Haveg 41, Lead, Glass, Hastelloy B or H2O – 50% H2SO4 <60°C – Steel Saran [illegible] Lined Pipe and Valves. High Stage Conc. to 70% H2SO4 Hot (150–180°C) Haveg 41, [illegible] Glass or Lead Clad Steel Pipe – Valves Haveg 41, Glass, Lead, Alloy 20, Durimet 20 – Cold 70% [illegible] <60°C Steel – Saranlloy Hypalon. Pipe and Valves [illegible] of FA 20 Alloys. All Tanks to have a Drain to Sewer for Tank Cleaning.

WASTE ACID
RECOVERY PLANT FLOW SHEET
70% H2SO4 – 50 Tons/Day [illegible]
[illegible] 10/22/5[illegible]